NASA TECHNICAL MEMORANDUM

NASA TM X-52132

NASA TM X-52132

GPO PRICE $ ____

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Hard copy (HC) $1.00

Microfiche (MF) .50 ¢

ff 653 July 65

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N65-34247

N65-34247 (ACCESSION NUMBER)	(THRU)
22 (PAGES)	1 (CODE)
TMX-52132 (NASA CR OR TMX OR AD NUMBER)	17 (CATEGORY)

FACILITY FORM 602

by Marvin Garfinkle

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Cleveland, Ohio

TECHNICAL PREPRINT prepared for Fourth Symposium on Refractory Metals sponsored by the Refractory Metals Committee of the Institute of Metals Division of AIME, French Lick, Indiana, October 3-5, 1965

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION · WASHINGTON, D.C. · 1965

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E-3124

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INTRODUCTION

The hardness of polycrystalline tungsten at room temperature has been observed to be decreased substantially by the addition of approximately 5 a/o (atomic percent) rhenium (refs. 1 and 2). Observation of this effect has prompted several investigators (refs. 2 to 4) to examine the tensile properties of dilute polycrystalline tungsten-rhenium alloys to determine whether a corresponding increase in ductility occurs. Increased ductility has been observed in both worked and recrystallized material. A maximum in ductility occurs at approximately 5 a/o rhenium.

The particular manner in which rhenium additions affect the mode of deformation of tungsten has not been well documented. In fact, uncertainties still exist as to the slip systems responsible for deformation in unalloyed tungsten single crystals.

Although it is generally agreed that the slip direction in bcc metals is ⟨111⟩, there are various points of view as to the slip plane (refs. 5 and 6). Garlick and Probst (ref. 7) have shown that slip in tungsten occurs on {112} planes in ⟨100⟩ oriented single crystals at room temperature, but slip lines were observed only after 10 percent elongation. The tensile curve for the ⟨100⟩ oriented crystals showed no yield point, with plastic flow being evident shortly after loading.

The present study was initiated to determine the effects of rhenium on the room-temperature deformation behavior of ⟨100⟩ oriented tungsten single crystals.

MATERIALS AND EXPERIMENTAL PROCEDURE

Single crystals, nominally 1/8 inch in diameter and containing 0, 3, 5, 7 and 9 a/o rhenium, were prepared by zone melting of sintered and swaged powder compacts by utilizing the electron-beam apparatus described by Witzke (ref. 8). The desired orientation was produced by growth onto ⟨100⟩ oriented seed crystals of unalloyed tungsten.

The orientations of all single crystals were determined by Laue back-reflection techniques to be within 4° of the [001] direction. Typical impurity levels of single crystal rods are presented in table I. The rhenium contents of the crystal rods were within 0.5 percent of their nominal values.

These crystals were cut and centerless ground to button-head specimens with a reduced section 1.125 inch in length and 0.085 inch in diameter. On several specimens, 1/2-inch long flats were ground in the reduced section parallel to specific crystallographic planes. The orientations of these flats were within 4° of the desired crystallographic planes. The worked surface layer of all specimens was removed by electropolishing in an NaOH solution to a depth of at least 0.005 inch.

Several tensile specimens of each composition (without flats) were fitted with foil strain gages having a gage length of 0.035 inch. Two gages were mounted at the midpoint of each specimen on opposite sides of the reduced section.

The tensile specimens were loaded on an Instron Universal Testing Machine. Strain was monitored through an external bridge circuit in which the specimen strain gages comprised two arms of the bridge. The bridge output was recorded against the applied stress.

RESULTS

The room-temperature tensile behavior of the ⟨100⟩ oriented tungsten and tungsten-rhenium single crystals is summarized by the graphs of true stress plotted against true strain in figure 1. Localized deformation was not observed until at least 3-percent strain.

The addition of rhenium had four distinct effects on the tensile behavior of tungsten single crystels:

(1) Rhenium increased the proportional limit stress. This effect is illustrated in figure 2. Because the tensile curves of the 0 and 3 a/o alloys did not show an abrupt transition from linear behavior, the proportional limit stresses of these alloys were determined by repeated reloading to successively higher stresses until nonlinear tensile behavior was observed.

(2) Rhenium promoted discontinuous yielding behavior at levels of 5 a/o and greater. The 5 a/o rhenium specimen exhibited elastic behavior until plastic flow began abruptly at an essentially constant stress. Some 5 a/o rhenium specimens showed a small yield-point drop. The magnitude of the yield-point drop increases with increasing rhenium content above 5 a/o. When unloaded and immediately reloaded after yielding, a 9 a/o rhenium crystal did not exhibit a yield-point drop but began to yield at the maximum stress to which it had been previously loaded. After aging at 1000° C for 1/2 hour, in a vacuum of $\sim 10^{-5}$ torr, the yield-point drop reappeared on reloading. An unalloyed specimen, similarly strained and aged, did not show a yield-point drop.

(3) The flow stress and work hardening rate decreased with increasing rhenium content. Figure 3 illustrates the stress in excess of the proportional limit stress necessary to continue plastic flow.

(4) Rhenium increases the reduction in area at fracture of ⟨100⟩ oriented crystals, as is illustrated in figure 4. All the crystals, regardless of rhenium content, failed by cleavage. From macroscopic examination, the cleavage plane appeared to be the (001) plane. The cross sections of the unalloyed and the 3 a/o rhenium crystals at failure were essentially circular. The cross sections of the 5, 7, and 9 A/o rhenium crystals, however, were elliptical.

The elliptical cross sections exhibited by the alloy specimens resulted from highly localized deformation, which began at approximately 3 percent elongation.

The 5 a/o rhenium specimen necked and then failed in the necked region. The 9 a/o rhenium specimen however, necked to an essentially constant cross section, the length of which increased at the expense of the remaining circular cross-sectional material. The axes of the elliptical cross sections were always in ⟨110⟩ directions.

Localized necking in the 9 a/o rhenium crystal often occurred in more than one region, and the necked portions finally joined to produce an elliptical cross section in the entire reduced section. In some cases, the major axes of the two necked regions were 90° apart, so that they could not join. Such a specimen is illustrated in figure 5. Once such a neck had formed, both the circular and the elliptical regions of the 9 a/o rhenium specimens remained essentially constant in cross-sectional area during further plastic straining. The overall straining was accommodated by an increase in the length of the elliptical region at the expense of the circular region. Because the load during plastic flow was constant, the stresses in the circular and the elliptical regions were also constant and were calculated to be 74,000 and 141,000 psi, respectively.

An analysis of the asterism exhibited by a ⟨110⟩ Laue back-reflection pattern of the elliptical region on the 9 a/o rhenium specimen indicates that the crystallographic planes rotate about the ⟨110⟩ axis during deformation. Total rotation of the tensile axis in this region was approximately 4°.

Slip behavior was studied on the 0, 5, and 9 a/o rhenium tensile specimens by examining slip traces on the (100) and $(1\bar{1}0)$ oriented flats after straining in approximately 1-percent increments. Before reloading, the specimens were electrolytically repolished. This process was continued to fracture.

No slip traces were observed on the unalloyed tungsten specimens even after straining to failure.(~14 percent total elongation). This observation is similar to that in previous work (ref. 7), where traces were not observed until 10-percent strain on ⟨100⟩ oriented tungsten crystals.

In view of this inability to observe slip traces on the as-deformed surfaces, an unalloyed tungsten crystal which had been strained 14 percent was etched and examined to see whether etching might reveal any evidence of the deformation mode. The etching revealed bright line traces, apparently corresponding to slip traces, which had not been visible on the as-deformed surface. On this particular specimen, the flats were parallel to {110} planes, 90° apart. Two sets of lines were evident on each flat, one set between 51° and 57° and the other set between 123° and 129° to the tensile axis. These lines correspond to slip on {110} planes.

On a second unalloyed tungsten specimen, strained only 1 percent and similarly etched, etch pits were observed which defined a lamellar pattern (fig. 6). The lamellae observed on the {110} face were 54° to the tensile axis.

The {110} face was uniformly etched. If these lamellae correspond to slip traces in a manner analogous to the finding of Low and Guard (ref. 9) in silicon iron, then slip has occurred on a plane of the ⟨111⟩ zone. This zone includes both {110} and {112} planes.

On another unalloyed specimen, also strained 1 percent and etched, etch pit lines were observed on a {100} face. These lines were at angles of 25°, 42°, 61°, and 87° to the tensile axis. The {112} planes of the ⟨111⟩ zone intersect the {100} face at 26.6° and at 63.4°, while the {110} planes of this zone intersect the {100} face at 45° and at 90°. This indicates that both {112} and {110} are operating slip planes in unalloyed tungsten crystals.

In contrast to the behavior observed in unalloyed tungsten, slip traces were readily visible on the unetched 5 a/o rhenium alloy after only 1-percent strain. The slip traces on the {110} flat were 90° to the tensile axis and quite wavy. The traces on the {100} flat were straight and at an angle of 61° to 67° with the tensile axis. These observations are consistent with slip on {112} planes. In some regions, $(\bar{1}12)$ slip predominated, while in other regions $(1\bar{1}2)$ slip was more apparent. In intermediate regions, both slip planes were equally operative.

Essentially the same traces were visible in this alloy after 4-percent strain, with the traces being more distinct in the necked portions of the specimen. Traces attributable to {110} slip were found only in the necked region adjacent to the failure.

Slip traces were distinct on the unetched 9 a/o rhenium specimen over the entire reduced section after 1-percent elongation. As in the case of the 5 a/o rhenium specimens, traces on the (110) flat were 90° to the tensile axis and quite wavy. On the (010) face, the traces were at angles of 62° and 118° to the tensile axis, indicating both $(\bar{1}12)$ and $(1\bar{1}2)$ slip. Traces attributable to {110} slip were visible along the elliptical necked region only.

The metallography of these specimens is discussed in more detail in reference 10.

DISCUSSION

The most noticeable effect of rhenium on the tensile behavior of ⟨100⟩ oriented tungsten single crystals was the decrease in work-hardening rate with increasing rhenium content. Discontinuous yielding was observed at 5 a/o rhenium and higher and the primary mode of deformation became {112} slip.

The processes involved in the deformation of ⟨100⟩ oriented unalloyed tungsten crystals have not been well documented because slip traces are not observable until considerable deformation has occurred. The etch pit pattern observed in the present study after 1-percent elongation can be attributed to both {110} and {112} slip. Thus slip in unalloyed tungsten single crystals can be attributed to both {112}⟨111⟩ and {110}⟨111⟩ slip systems, which possibly leads to dislocation interactions that cause the high rates of work hardening observed.

The high degree of work hardening evident in unalloyed ⟨100⟩ oriented tungsten single crystals may explain the difficulty in observing slip traces in the early stages of deformation. The amount of slip that can occur in any localized region may be so small that slip bands intersecting the surface are too narrow to be observed.

Work hardening is less evident for single crystals containing rhenium and is apparently virtually absent at a rhenium content of 9 a/o during uniform deformation.

Deformation in the 9 a/o rhenium alloy occurs by {112} ⟨111⟩ slip. The slip direction was confirmed by the asterism exhibited by the ⟨110⟩ Laue pattern of the deformed region, which indicates bending of crystallographic planes about a ⟨110⟩ axis. The slip direction must be perpendicular to this axis of bending and in the slip plane (ref. 11). Only ⟨111⟩ slip meets these conditions.

During necking, which started at ~3 percent strain, slip on {112} planes of the 9 a/o rhenium crystals was localized in the region joining the circular and elliptical cross section of the specimen, which caused the elliptical region to increase in length. Since the tensile flow stress did not vary during plastic deformation, the resolved shear stress to sustain slip on the {112}⟨111⟩ system can be calculated as 35,000 psi. The resolved shear stress on the same {112}⟨111⟩ systems in the necked region was calculated to be much higher, 66,500 psi. Because {110} slip traces were visible only in the work-hardened elliptical necked region, slip on this plane apparently is suppressed in rhenium-containing crystals until a relatively high shear stress is achieved.

Discontinuous yielding in ⟨100⟩ oriented crystals first became evident at a rhenium content of approximately 5 a/o. Both the yield-point drop and the previously discussed decrease in work hardening became more pronounced with increasing rhenium content up to at least 9 a/o, which suggests that these observed phenomena are probably related. In addition, the work-hardening behavior of the ⟨100⟩ oriented 9 a/o rhenium crystals was similar to that previously observed for unalloyed ⟨110⟩ oriented tungsten single crystals before necking (ref. 7), as is illustrated in figure 7. Both materials show upper yield points. Furthermore, ⟨110⟩ oriented unalloyed crystals neck to an essentially elliptical cross section with the major axes in the ⟨110⟩ direction (ref. 12), as do the 9 a/o rhenium crystals. This, along with asterism studies of the necked region of a ⟨110⟩ oriented unalloyed crystal for which the author thanks R. G. Garlick of the Lewis Research Center, indicated that they both deform by slip on the {112} plane. The flow stress of the ⟨110⟩ oriented unalloyed tungsten crystal is, however, considerably higher than that of the ⟨100⟩ oriented 9 a/o rhenium crystals.

Any mechanism of deformation speculated upon to explain the observed behavior must account for both the orientation and the rhenium effects. When pulled in the ⟨110⟩ direction, a tungsten single crystal exhibits a yield point, and subsequent plastic flow is primarily by slip on {112} planes. Such a yield point and slip system was not observed by crystals pulled in the ⟨100⟩ direction unless they contained greater than 5 a/o rhenium in solid solution. The proportional limit stress of the rhenium-containing crystals was greater than that of the unalloyed ⟨100⟩ oriented crystals. The proportional limit stress of the ⟨110⟩ oriented unalloyed crystals was probably greater than that of the ⟨100⟩ oriented crystals because their observed yield point occurred at a considerably greater stress than the initial flow stress of the ⟨100⟩ oriented crystals.

Snoek (ref. 13) has suggested that an applied stress that gives rise to a nonspherically symmetric lattice distortion can cause a redistribution of interstitials to particular sites and thus reduce the strain energy of the lattice. Furthermore, Nabarro (ref. 14) has pointed out that the stress field associated with a distorted lattice can strongly interact with the shear stress component of a dislocation stress field. Such an interaction could cause a redistribution of interstitials in the stress field of the dislocation.

Schoeck and Seeger (ref. 15) have suggested that such a redistribution can reduce the strain energy of the dislocation.

If the applied stress considered is a tensile stress, the resulting lattice distortion will depend on the crystallographic direction of this stress, and thus, so would also the distribution of interstitials in the stress field of the dislocation.

If the applied stress is in the $\langle 100 \rangle$ direction, the resulting interstitial distribution might have only a negligible effect on the strain energy of dislocations, and thus permit dislocation motion at a relatively low stress level on both $\{110\}$ and $\{112\}$ planes with a resulting high rate of work hardening.

Alternatively, the interstitial distribution resulting from a $\langle 110 \rangle$ tensile stress might significantly decrease the strain energy of the dislocations and restrict their motion, which would thus prevent plastic flow until dislocation multiplication occurs primarily on $\{112\}$ planes.

The addition of rhenium to tungsten may affect interstitial-dislocation interactions, possible the distribution of interstitials at dislocations. That rhenium can affect interstitial-dislocation interactions is suggested by the aging experiments. While a strained unalloyed $\langle 100 \rangle$ oriented crystal does not exhibit a yield-point drop after aging, a similar specimen, similarly strained and aged, containing 9 a/o rhenium, does.

If such a redistribution significantly decreases dislocation strain energy, dislocation motion can be restricted. This restriction would prevent plastic flow until dislocation multiplication occurs on only the $\{112\}$ plane, which would result in a yield-point drop. Plastic flow could then occur with little work hardening.

CONCLUDING REMARKS

The addition of rhenium to ⟨100⟩ oriented tungsten single crystals has a significant effect on deformation behavior.

(1) Rhenium additions both increased the proportional limit stress and decreased the flow stress of tungsten single crystals. While work hardening is appreciable in unalloyed tungsten, it is essentially nonexistent in the 9 a/o rhenium alloy until necking occurs.

(2) Discontinuous yielding occurs at 5 a/o rhenium, with the magnitude of the yield-point drop increasing with increasing rhenium content. The yielding and flow behavior of the 9 a/o rhenium ⟨100⟩ oriented crystals is similar to that exhibited by unalloyed ⟨110⟩ oriented tungsten single crystals but at a much lower stress level.

(3) Alloys with 5 a/o or more rhenium neck before failure; the cross section of the necked region is elliptical with axes in the ⟨110⟩ direction. This deformation process involves bending of crystallographic planes about the ⟨110⟩ axis. Similar behavior has been observed in the deformation of ⟨110⟩ oriented unalloyed tungsten crystals.

(4) While deformation of unalloyed crystals apparently involves slip on both {112} and {110} in the ⟨111⟩ direction, the addition of rhenium promoted {112}⟨111⟩ slip. Faint {110} slip traces were observed in the rhenium alloys only during necking. The 9 a/o rhenium alloy deformed uniformly only by {112} ⟨111⟩ slip; {110} slip traces were observed only in the elliptical necked region. This observation suggests that work hardening in tungsten may be associated with {110} slip.

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(5) A possible mechanism is proposed to elucidate both the effect of dilute rhenium additions observed in the present study and the orientation effect observed previously in unalloyed tungsten. Essentially, these observed phenomena are interpreted in terms of the orientation dependence of the sites occupied by interstititials in the stress field of dislocations when the lattice is elastically deformed and in terms of the effect of rhenium additions on the distribution of interstitials in tungsten.

REFERENCES

1. Allen, B. C.; and Jaffee, R. I.: ASM Trans., 1963, vol. 56, p. 395.
2. Pugh, J. W.; Amra, L. H.; and Hurd, D. T.: ASM Trans., 1962, vol. 55, p. 451.
3. Ratliff, J. L.; Maykuth, D. J.; Ogden, H. R.; and Jaffee, R. I.: Trans. Met. Soc. AIME+ 1964, vol. 230, p. 490.
4. Raffo, P. L.; Klopp, W. D.; and Witzke, W. R.: NASA Technical Note D-2561, Jan. 1965, p. 16.
5. Hoke, H. J.; and Maddin, R.: J. Mech. Phys. Solids, 1956, vol. 5, no. 1, p. 26.
6. Leber, S.; and Pugh, J. W.: Trans. Met. Soc. AIME, 1960, vol. 218, p. 791.
7. Garlick, R. G.; and Probst, H. B.: Trans. Met. Soc. AIME, 1964, vol. 230, p. 1122.
8. Witzke, W. R.: Zone Melting of Tungsten by Electron Bombardment. Proc. First Symposium on Electron-Beam Melting, Boston (Mass.), Mar. 20, 1959, p. 73.
9. Low, J. R.; and Guard, R. W.: ACTA Met., 1959, vol. 7, p. 171.
10. Garfinkle, M.: Proposed NASA Technical Note.

11. Maddin, R.; and Chen, N. K.: Geometrical Aspects of the Plastic Deformation of Metal Single Crystals, Progress in Metal Physics, vol. 5, p. 53, Pergamon Press, London, 1954.

12. Rose, R. M.; Ferriss, D. P.; and Wulff, J.: Trans. Met. Soc., AIME, 1962, vol. 224, p. 985.

13. Snock, J. L.: Physica, 1941, vol. 8, p. 731.

14. Nabarro, F. R. N.: Report of Conf. on Strength of Solids, the Physical Socieyt, London, 1948, p. 38.

15. Schoeck, G.; and Seeger, A.: Acta Met., 1959, vol. 7, p. 469.

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TABLE 1

TYPICAL IMPURITY LEVELS IN TUNGSTEN

SINGLE CRYSTAL RODS

Element	Amount, ppm	Element	Amount, ppm
Al	< 0.01	Mo	2
Be	.001	N	5
C	3	Nb	< 1
Ca	.1	Ni	0.2
Co	.02	O	5
Cr	.3	Si	< 0.1
Cu	.09	Ta	< 3
Fe	.2	Ti	0.01
H	< 1	V	0.05
Mg	<.01	Zr	0.01

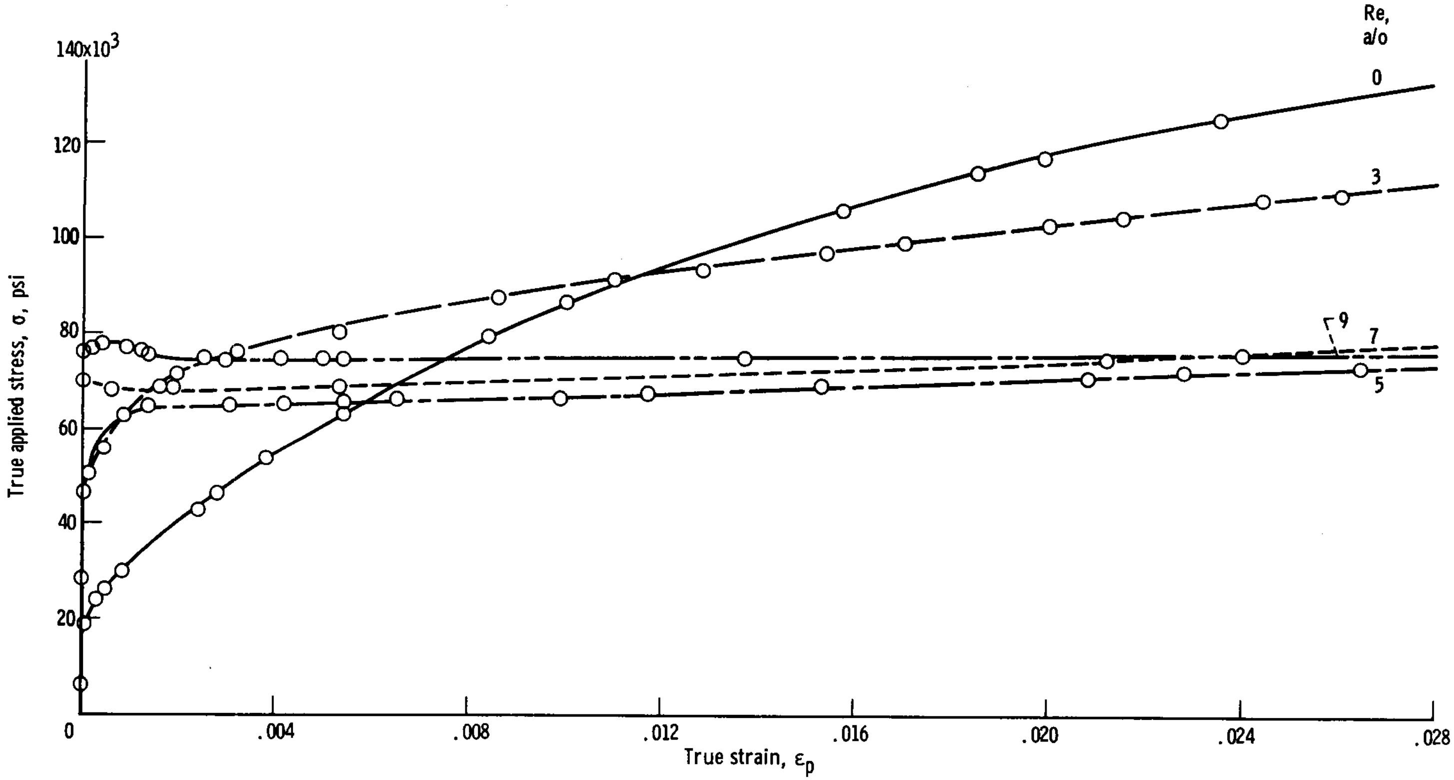

Figure 1. - Tensile curves of ⟨100⟩ oriented tungsten single crystals containing various rhenium contents. Beyond 0.030 strain localized deformation occurs in the 5, 7, and 9 a/o rhenium specimens.

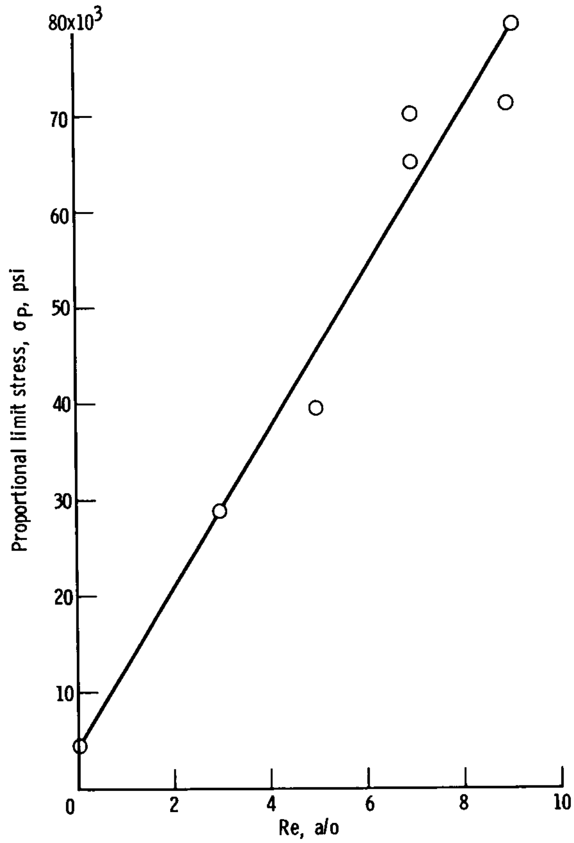

Figure 2. - Effect of rhenium on proportional limit stress of ⟨100⟩ oriented tungsten single crystals.

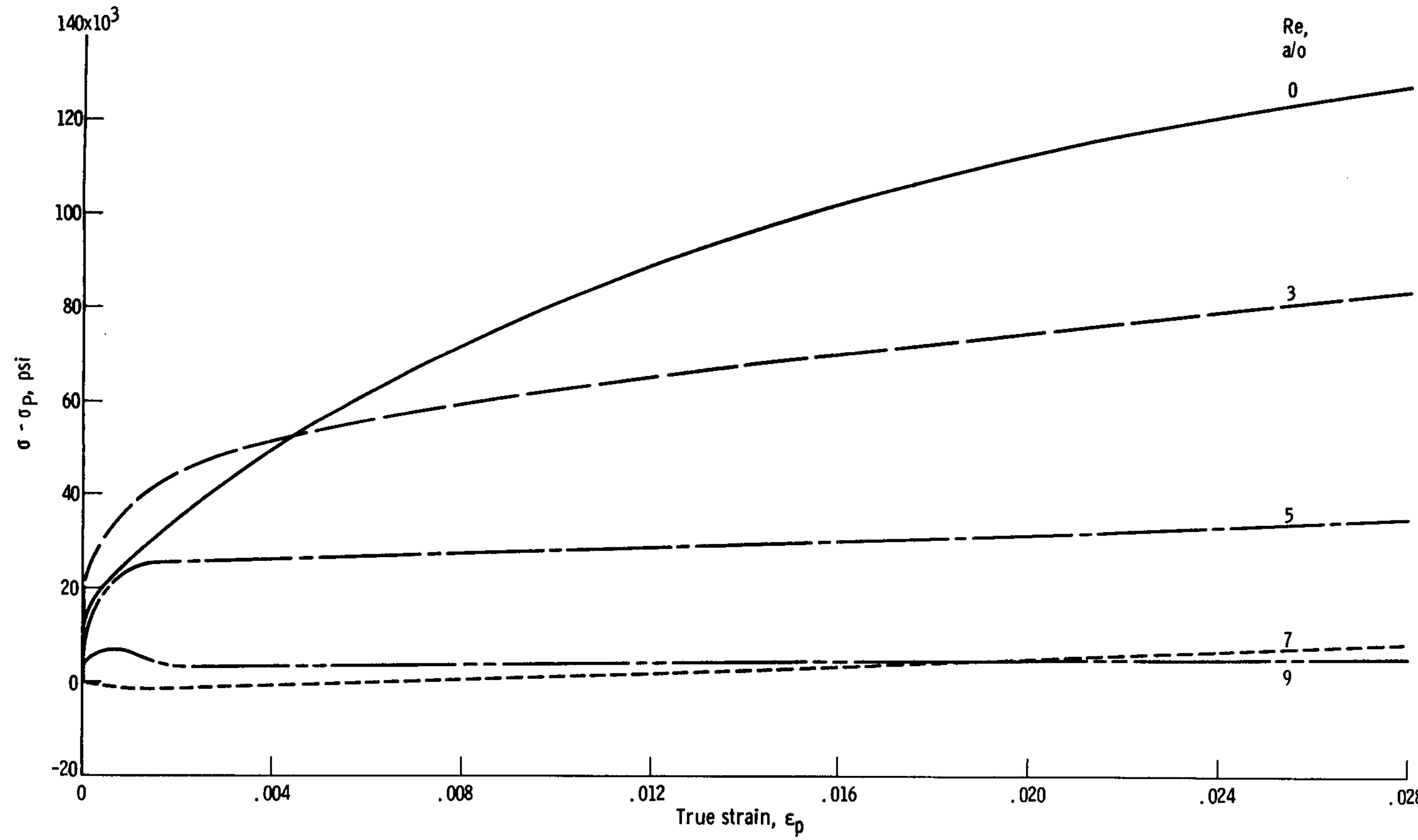

Figure 3. - Effect of rhenium on flow stress in excess of proportional limit stress of ⟨100⟩ oriented tungsten single crystals.

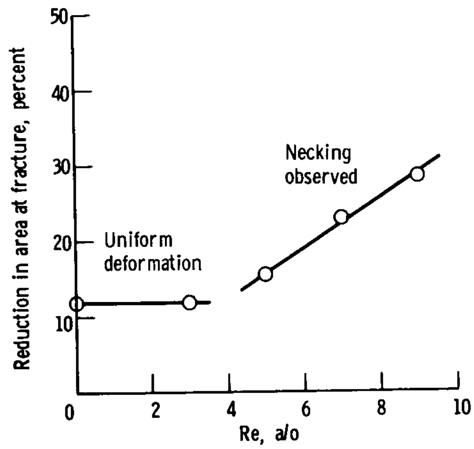

Figure 4. - Effect of rhenium on reduction in area of ⟨100⟩ oriented tungsten single crystals.

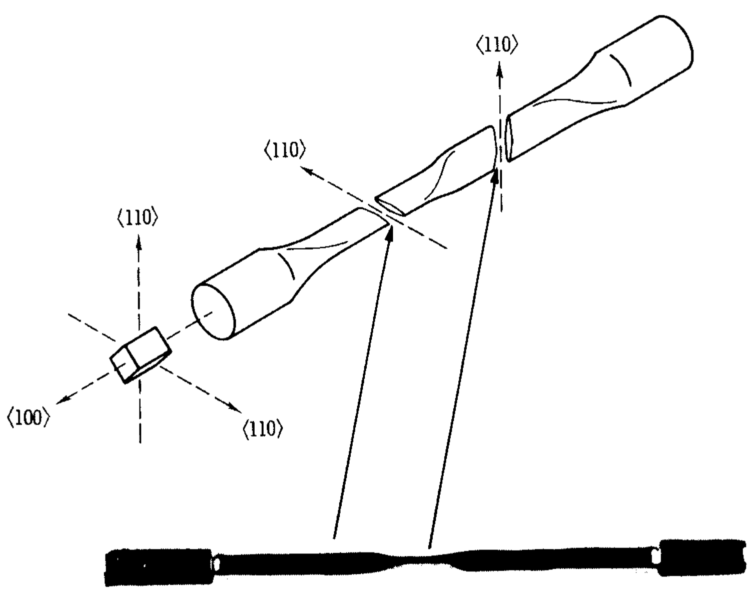

Figure 5. - Tungsten - 9% rhenium tensile specimen after 15% strain.

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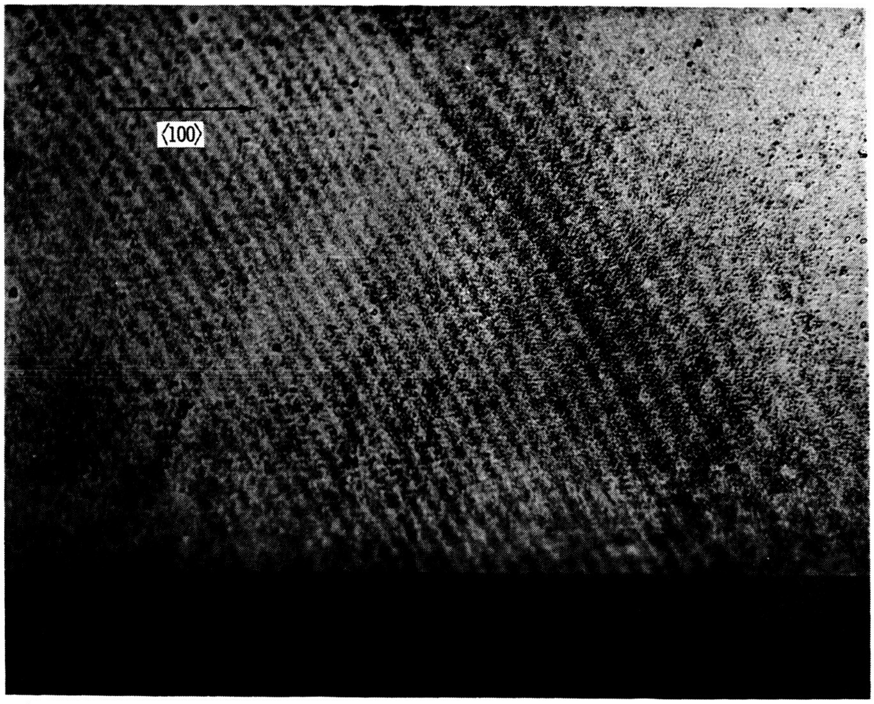

Figure 6. - Etch pit pattern on {110} flat of ⟨100⟩ oriented trunsten single crystal after 1% elongation. Etchant, $CuSo_4 + NH_4OH$. X250.

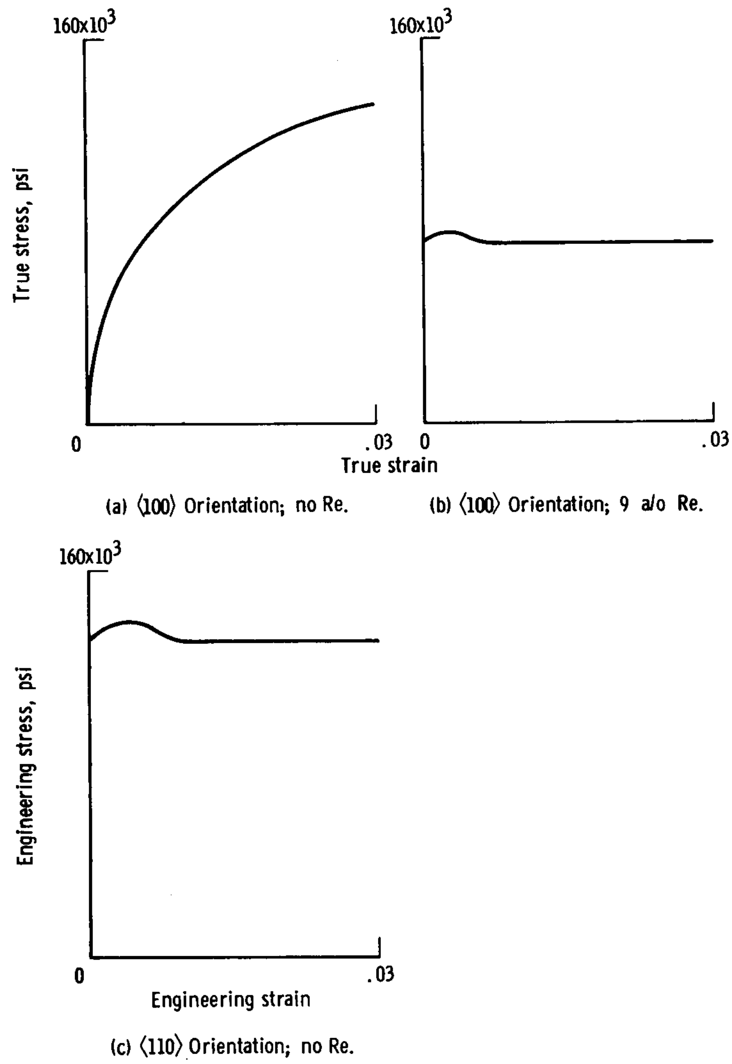

Figure 7. - Effect of orientation and rhenium content on tensile behavior of tungsten single crystals.